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2. REPORT DATE

August 1993

3. REPORT TYPE AND DATES COVERED

Technical (6/1/93-5/31/94)

4. TITLE AND SUBTITLE

The Ferroelectric Behavior of Odd-Numbered Nylons

5. FUNDING NUMBERS

N00014-91-J-1078

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8. PERFORMING ORGANIZATION
REPORT NUMBER

#29

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Dr. JoAnn Milliken
Office of Naval Research
Arlington, VA 22217-5000

10. SPONSORING / MONITORING
AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

Submitted to "Ferroelectrics"

DTIC
ELECTE
APR 12 1994
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12a. DISTRIBUTION / AVAILABILITY STATEMENT

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248V 94-11024

13. ABSTRACT (Maximum 200 words)

The combined results of the present investigation of Nylon 5 and Nylon 9 and the previous investigation of Nylon 11 and Nylon 7 reveal that the odd-numbered nylons exhibit classic ferroelectric J-E and D-E hysteresis behavior at room temperature. In addition, the remanent polarization, P_r , of Nylon 5 and Nylon 9 are found to be $125 \sim 130$ mC/m² and $65 \sim 70$ mC/m², respectively. The remanent polarization of the odd nylon series is observed to increase linearly with increasing dipole density as does the melting point. The coercive field also shows a linear increase with dipole density. The present study also shows that both uniaxially oriented and unoriented odd-numbered nylons exhibit ferroelectric hysteresis behavior, and that the ferroelectric response can be significantly enhanced by uniaxial orientation.

14. SUBJECT TERMS

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION
OF REPORT

UNCLASSIFIED

18. SECURITY CLASSIFICATION
OF THIS PAGE19. SECURITY CLASSIFICATION
OF ABSTRACT

20. LIMITATION OF ABSTRACT

OFFICE OF NAVAL RESEARCH

Contract N00014-91-J-1078

R&T Code 4132046

Technical Report No. 29

THE FERROELECTRIC BEHAVIOR OF ODD-NUMBERED NYLONS

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Prepared for Publication
in
Ferroelectrics

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August 1993

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THE FERROELECTRIC BEHAVIOR OF ODD-NUMBERED NYLONS

B.Z. MEI, J.I. SCHEINBEIM AND B.A. NEWMAN

SYNOPSIS

The combined results of the present investigation of Nylon 5 and Nylon 9 and the previous investigation of Nylon 11 and Nylon 7 reveal that the odd-numbered nylons exhibit classic ferroelectric J - E and D - E hysteresis behavior at room temperature. In addition, the remanent polarization, P_r , of Nylon 5 and Nylon 9 are found to be 125~130 mC/m² and 65~70 mC/m², respectively. The remanent polarization of the odd nylon series is observed to increase linearly with increasing dipole density as does the melting point. The coercive field also shows a linear increase with dipole density. The present study also shows that both uniaxially oriented and unoriented odd-numbered nylons exhibit ferroelectric hysteresis behavior, and that the ferroelectric response can be significantly enhanced by uniaxial orientation.

INTRODUCTION

Poly(vinylidene fluoride) (PVF₂)¹ and its copolymers² were the only known class of ferroelectric polymers until our discovery that melt-quenched and cold-drawn Nylon 11 and Nylon 7 were also ferroelectric polymers exhibiting similar piezoelectric activity^{3,4}. Although large piezoelectric and pyroelectric coefficients have been measured on several polymer electrets such as amorphous poly(vinyl chloride)⁵ and the amorphous copolymer of vinylidene cyanide-vinyl acetate⁶, it is commonly understood

that in these amorphous systems field induced dipole orientation can be produced at temperatures equal to or greater than T_g , and "frozen in" by cooling under field. These materials are of course not ferroelectric. Earlier work on the semicrystalline polymers, poly(vinyl fluoride)⁷ and Nylon 11⁸, showed that these polymers can be poled to give a measurable piezoelectric or pyroelectric response, but no experimental evidence was presented in these earlier studies to demonstrate the reversible polarization found in ferroelectric materials. Our work on Nylon 11 and Nylon 7 revealed for the first time that these polymers exhibit J - E and D - E hysteresis behavior, typical of classic ferroelectric materials, and further X-ray diffraction studies combined with FTIR studies confirmed that this behavior was associated with switching of molecular dipoles. This work then led to speculation about whether the other odd-numbered nylons, such as Nylon 5, Nylon 9, etc., are also ferroelectric and how large their piezoelectric activities might be.

These other odd-numbered nylons exhibit an isomorphous relationship with the crystal structures observed in the previously studied odd-numbered nylons. Polyamides from ω -amino acids with even numbers of CH_2 groups are conventionally described as odd-numbered nylons, and it is commonly accepted that polyamide molecules usually tend to array themselves with a maximum degree of coupling (hydrogen bonding) between amide groups on adjacent chains during crystallization^{9,10}. This forms the basis for the so-called hydrogen-bonded sheet structures. In 1959, Slichter¹¹ proposed a triclinic unit cell for nylon 11 with one chain per unit cell, with the chain possessing an all-trans conformation giving rise to a net dipole moment in the unit cell. We also note that for the odd-numbered nylons series, decreasing the number of CH_2 groups increases the concentration of amide groups thereby increasing the dipole density. In a recent study, we reported that the ferroelectric response of both Nylon 11 and Nylon 7 originated in the crystalline

regions^{12,13}. This fact led us to assume that if all the odd-numbered nylons possessing similar crystal structures were ferroelectric, then given the appropriate sample preparation and electroprocessing, the remanent polarization of the odd nylons should increase down the series. In other words, the lower-order odd nylons should exhibit larger remanent polarizations. In addition, we observe that the relationship between the remanent polarization and the dipole density seems to bear a close resemblance to the relationship between the melting points and the number of amide groups, which was previously observed^{9,14,15}. Thus, the correlation between remanent polarization and melting point for the odd nylons is significant since it indicates both the origin of the ferroelectric response and the determining factor of the melting points in the series of odd-numbered nylons.

EXPERIMENTAL

The nylon films used in this study were prepared by melting nylon powder between aluminum foils in a hot press at 300°C and 230°C for Nylon 5 and Nylon 9, respectively, and pressing into thin films. The molten films were then quenched in an ice bath. The quenched films were uniaxially cold-drawn at room temperature to a draw ratio of 3:1. The stretched films had a thickness of 10~20 microns.

Gold electrodes (area of 10~20 mm²) were evaporated on opposing sides of the films. The samples were always dried and stored under high vacuum conditions in order to minimize moisture absorption. Samples were pretreated before poling by application of a static electric field for the purpose of field-sweeping out most mobile ionic impurity species, which dramatically reduced the ionic conduction observed during the following poling procedure. Samples were placed in a silicone oil bath and poled by applying a triangular shaped electric field pulse with a maximum amplitude

of 150~200 MV/m at room temperature. The period of the triangular voltage waveform was 640s.

Nylon 11 and Nylon 7 samples were also prepared and poled using the methods described above for the purpose of comparing the ferroelectric behavior of all these odd nylons.

RESULTS

Figure 1(a) shows the measured current density, J , versus the electric field, E , for melt-quenched and cold-drawn Nylon 11, Nylon 9, Nylon 7 and Nylon 5 films. By integrating the current density with respect to time, the typical D - E hysteresis loops, shown in Figure 1(b), were obtained. Apparently, both Nylon 5 and Nylon 9 also exhibit the current density peak previously observed in Nylon 11 and Nylon 7 which indicates a polarization reversal phenomenon. The apparent coercive fields were determined by the value of the field where the polarization passes zero are usually found to be 100 and 75 MV/m for Nylon 5 and Nylon 9, respectively. The value of measured remanent polarization, P_r , for Nylon 5 and Nylon 9 determined by the intercept of the hysteresis loops with the D -axis are 125~130 and 65~70 mC/m² respectively. Table 1 shows the values of the remanent polarization, P_r , and the coercive fields, E_c , for Nylon 11, Nylon 9, Nylon 7 and Nylon 5. Comparing the values of P_r of the four odd nylons, it is obvious that Nylon 5 possesses the highest remanent polarization; in addition, for the odd-numbered nylon series studied, the remanent polarization increases linearly with increasing dipole density as expected. In other words, the remanent polarization of the odd-numbered nylons does increase with decreasing numbers of CH₂ groups.

It was found during the poling procedure that both the ionic conduction and apparent coercive fields measured for the lower-order odd nylons are strongly affected by moisture absorption. Compared to the higher-order odd nylons, Nylon 5 is much more sensitive to moisture because of the higher density of hydrogen bonding amide groups in its molecular chains. The absorbed moisture results in very high ionic conduction. Usually, a dry-air purge can effectively reduce the ionic conduction during the poling process, but is found to increase the apparent coercive field by ~20%. This requires the application of much higher electric fields ($E_{max} = 200 \sim 250$ MV/m) to switch the dipoles.

Figure 2 shows the dependence of the coercive field, E_c , on the dipole density for the odd-numbered nylons. It is quite clear that the coercive fields for these four nylons increase successively, actually linearly, down the series, which indicates that it becomes more and more difficult for the polar amide groups to switch under the same applied electric field and the same experimental conditions, as the number of CH_2 groups decreases, while the number of hydrogen bonds per unit length of chain increases.

In Figure 3(a), the melting points of the odd-numbered nylons are plotted against their dipole density. The melting points are observed to increase linearly with increasing dipole density, which is in agreement with previous studies^{5,10,11}. Figure 3(b) shows the remanent polarization, P_r , versus the dipole density and indicates that P_r also increases linearly with increasing dipole density. It is interesting to note that P_r also increases linearly with increasing melting point as shown in Figure 4.

Since uniaxial stretching can significantly enhance the remanent polarization observed in ferroelectric polymers such as PVF_2 and melt-quenched Nylon 11 films¹⁶,

unoriented Nylon 7, and Nylon 5 films were examined. The results for both oriented and unoriented odd nylon samples are shown in Figure 5, and the values of P_r are listed in Table 2.

DISCUSSION

It is commonly accepted that the amide group has a large electric dipole moment with a magnitude of $\sim 3.7\text{D}^{17}$. Using the unit cell parameters proposed by Hasegawa et al¹⁸ for the odd-numbered nylon series, the theoretical dipole density can be computed by dividing the net dipole moment in the unit cell by the unit cell volume. This leads to a value of $1.34\text{ D}/100\text{\AA}^3$ for Nylon 11. By comparison, Nylon 9, Nylon 7 and Nylon 5 give values of 1.54, 2.06 and $2.94\text{ D}/100\text{\AA}^3$, respectively. Figure 3(b) shows that the bulk remanent polarization P_r is proportional to the theoretical dipole density P_s for the series of the odd-numbered nylons examined. Several theories have been proposed to model the piezoelectric and pyroelectric properties of polymers, the most detailed theory proposed being that of Wada and Hasegawa¹⁹, which models the polymer in terms of spheres (which are comprised of crystal lamellae with a surrounding oriented amorphous phase) with dielectric constant ϵ_c in a matrix with dielectric constant ϵ . These spheres are referred to as crystals even though they are in fact semicrystalline spherical aggregates. If the dipole moment density of these "crystals" is P_s , the bulk remanent polarization P_r is given by $P_r = fLP_s^{19}$, where $L = 3\epsilon/(2\epsilon + \epsilon_c)$ and f is the volume fraction of "crystals".

It should be noted that with this use of the word "crystal", the actual degree of crystallinity used in the conventional sense χ is less than f . However, if we assume that these spherical regions have similar structures for all the odd nylons examined, the

value of f can be estimated from $f = P_r / P_s \cdot 1 / L$. The value of the ratio P_r / P_s is found to be 1.24, 1.32, 1.29 and 1.27 for nylon 11, Nylon 9, Nylon 7 and Nylon 5, respectively, as given in Table 1. In the expression for $L = 3\epsilon / (2\epsilon + \epsilon_c)$, ϵ_c refers to the dielectric constant of the semicrystalline sphere. If we assume $\epsilon \gg \epsilon_c$, we obtain $L = 1.5$ and f can be estimated as 0.84, 0.89, 0.83 and 0.85 for Nylon 11, Nylon 9, Nylon 7 and Nylon 5, respectively. If we assume $\epsilon \approx \epsilon_c$, we obtain $L = 1$ and f can be estimated as approximately as 1.24, 1.32, 1.29 and 1.27. However it should be remembered that here f is not the volume fraction of crystals in the conventional sense. The degree of crystallinity will be less than f . If, for example, the degree of crystallinity for each sphere was 50%, the bulk degree of crystallinity would be $0.5f$. Finally, it should be noted that the model proposed by Wada and Hasegawa, although providing a theoretical framework which rationalizes why P_r should be linearly dependent on P_s , is not really appropriate for an oriented polymer which has been drawn 3:1. A more detailed analysis of the data requires a model which would more accurately represent the morphology of a drawn film.

The increasing apparent coercive fields for the odd-numbered nylons shown in Figure 2 may be interpreted as resulting from the increased amide content as the number of CH_2 groups decreases. As the density of hydrogen bonds per unit length of chain increases, the interaction between the molecular chains is enhanced, which in turn requires much higher energy to exceed the barrier to breaking and reforming the hydrogen bonded sheets which is the process by which dipoles switch into the direction of the external field.

With hydrophilic materials like the odd-numbered nylons, the water content is an additional complementary variable. The apparent coercive field was observed to be 100 MV/m for undried Nylon 5 samples, but increased to 120 MV/m when a dry-air

purge was used. Since it is generally believed that the absorbed water exists in the amorphous regions, the increase in the apparent coercive field may be explained on the basis of a two-phase model consisting of amorphous regions with dielectric constant ϵ and crystalline regions with dielectric constant ϵ_c . During the poling process, the dry-air purge can effectively reduce the moisture content of the amorphous regions. As a result, this decreases ϵ which in turn leads to a higher voltage drop across the amorphous regions. With the total voltage applied to the sample unchanged, the voltage drop across the crystalline regions will be decreased. In order to acquire the energy needed to obtain dipole switching in the crystalline regions, a higher external electric field is required and an increase in the apparent coercive field is observed.

Furthermore, if we assume that these four odd nylons possess approximately the same degree of crystallinity and the percentage of reorientable dipoles is close to the same value, the measured remanent polarizations should increase linearly with the dipole density. This is observed in the experimental results shown in Figure 3b. Since the moisture content and the degree of orientation both strongly affect sample crystallinity, it is very difficult to maintain close control over sample crystallinity during sample preparation. Consequently, it is somewhat arbitrary to assume, as we did, that all the melt-quenched nylon samples possess the same crystallinity. Although as previously discussed, the percentage of reoriented dipoles observed for the four nylons studied is very close to the same value, much more work is needed to clarify our understanding of the observed linear relationship between remanent polarization and theoretical dipole density.

It is well-known that the odd-numbered nylons show increased melting temperatures with increasing amide content of the molecules. The high melting temperatures shown by lower-order nylons are ascribed to the strong cohesion between

amide groups on neighboring chains. In Figure 4, it should be noted that if the curve is extrapolated to the point where P_r is equal to zero, the corresponding melting point is 410°K (137°C), which is approximately equal to the melting point of polyethylene, a pure hydrocarbon. This result may be explained by the relation $T_m = \Delta H_m / \Delta S_m$, where T_m is the melting temperature, ΔH_m and ΔS_m are the heat of fusion and the entropy of fusion, respectively. Itoh²⁰ pointed out in his paper published in 1976, that the hydrogen-bonded sheets in the crystalline regions of polyamides were found to be maintained after melting, so the higher melting points of lower-order odd nylons may be attributed to the smaller entropy of fusion, ΔS_m , because the chain configurations in the melt are more restricted in the odd nylons with higher amide content. On the other hand, the existence of the amide groups does not contribute significantly to the heat of fusion ΔH_m since the hydrogen bonded sheets remain after melting occurs. When the amide content is zero, which is the case for polyethylene, the entropy of fusion ΔS_m becomes much larger in melting due to the absence of the hydrogen bonds, so the melting temperature is much lower than that of the polyamides.

As we reported¹⁶, melt-quenched and cold-drawn Nylon 11 films exhibit much larger remanent polarizations than melt-quenched but unoriented samples; orientation increased the remanent polarization by almost 50%. A similar phenomenon was also observed for melt-quenched and unoriented Nylon 7 and Nylon 5 films. The values of P_r were enhanced by 40% for both nylons after uniaxial orientation. Since the ferroelectric response originates from the crystalline regions, these results may be attributed to the large increase in the order of the molecular arrangement in both crystalline and amorphous regions resulting from uniaxial orientation. The higher the orientation, the larger the number of dipoles in the crystalline regions which can be rotated about their chain axis by the external field and can contribute to the observed ferroelectric response.

Detailed studies on the polarization mechanisms, crystallite orientation, and the reorientation of the hydrogen bond dipoles (hydrogen bonded sheets) as result of a poling for the odd numbered-nylons will be deferred to a later publication.

CONCLUSIONS

Based on results of our studies on the ferroelectric behavior of both oriented and unoriented melt-quenched odd-numbered nylon films, the following conclusions are reached:

- (1) Melt-quenched and cold-drawn Nylon 9 and Nylon 5 films are ferroelectric.
- (2) The coercive field and remanent polarization of the odd-numbered nylons linearly increases with increasing (theoretical) dipole density .
- (3) The remanent polarization linearly increases with the increasing melting points of the odd -numbered nylons.
- (4) The unstretched melt-quenched odd-numbered nylons also show ferroelectric hysteresis behavior, but the ferroelectric response can be greatly enhanced by uniaxial drawing at room temperature.
- (5) The odd-numbered nylons constitute the second known class of ferroelectric polymers.

This work is supported by ONR, DARPA and CAFT.

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Figure 1(a). Current density, J , versus electric field, E , for as-stretched of (a) Nylon 11, (b) Nylon 9, (c) Nylon 7 and (d) Nylon 5.

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Figure 2. Dependence of coercive field, E_c , on the dipole density of odd-numbered nylons.

Figure 3(a). Dependence of melting point, T_m , on the dipole density of odd-numbered nylons.

Figure 3(b). Dependence of remanent polarization, P_r , on the dipole density of odd-numbered nylons.

Figure 4. Dependence of remanent polarization, P_r , on melting point, T_m , for the odd-numbered nylons.

Figure 5(a) Current density, J , versus electric field, E , for a. oriented Nylon 7, a'. unoriented Nylon 7, b. oriented Nylon 5 and b'. unoriented Nylon 5 films.

Figure 5(b). Electric displacement, D , versus electric field, E , for a. oriented Nylon 7, a'. unoriented Nylon 7, b. oriented Nylon 5 and b'. unoriented Nylon 5 films.

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Table 2. The remanent polarization of oriented and unoriented odd-numbered nylons.

Table 1. The Dipole Density, Remanent Polarization, Coercive Field, and the percentage of switched dipoles of Odd-Numbered Nylons

Odd Nylons	Nylon 11	Nylon 9	Nylon 7	Nylon 5
Dipole density	1.34D/100Å ³	1.54D/100Å ³	2.06D/100Å ³	2.94D/100Å ³
P_r (mC/m ²)	56	68	86	125
E_c (MV/m)	64	75	80	100
P_r/P_s	1.24	1.32	1.25	1.27

Table 2. The remanent polarization of oriented and unoriented odd nylons

	Nylon 11	Nylon 9	Nylon 7	Nylon 5
oriented	56	70	82	125
unoriented	25	--	50	78

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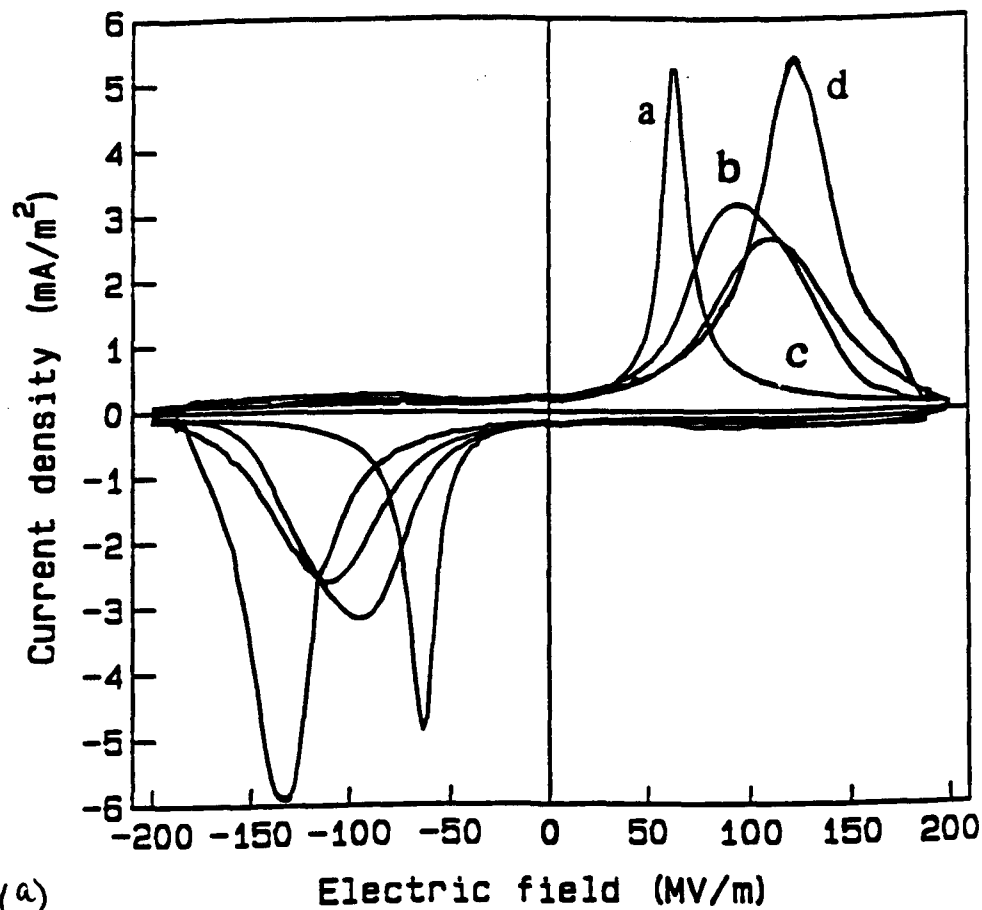


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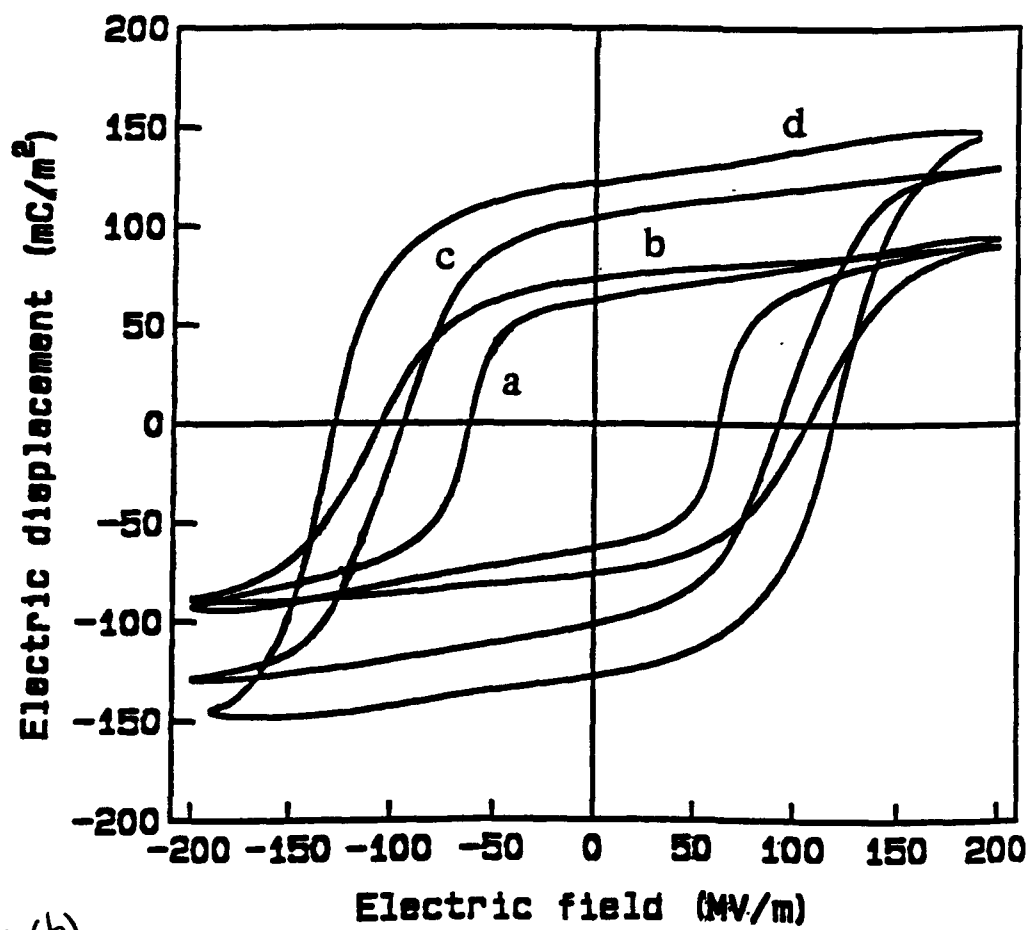


Fig-1 (b)

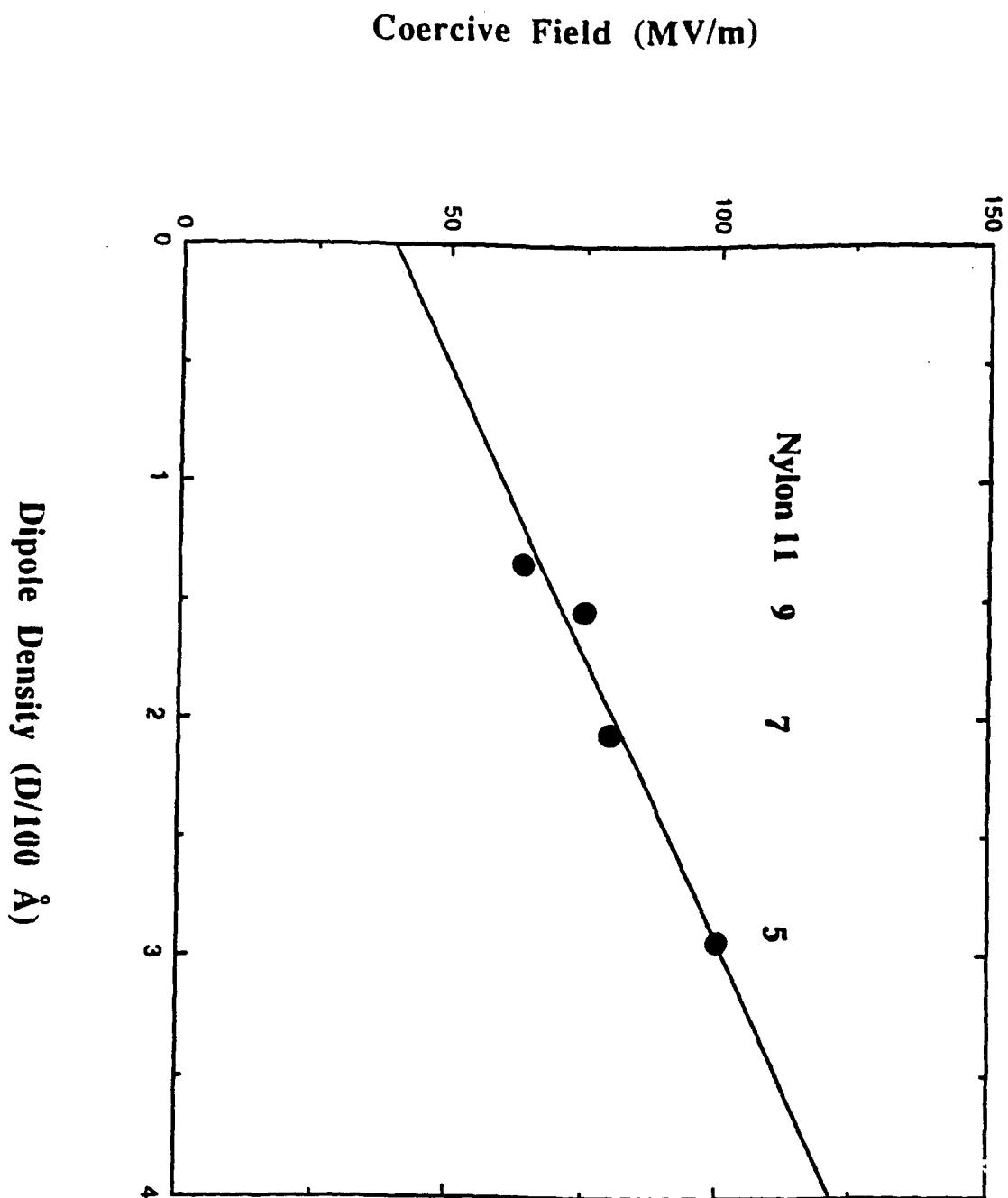


Fig. 2

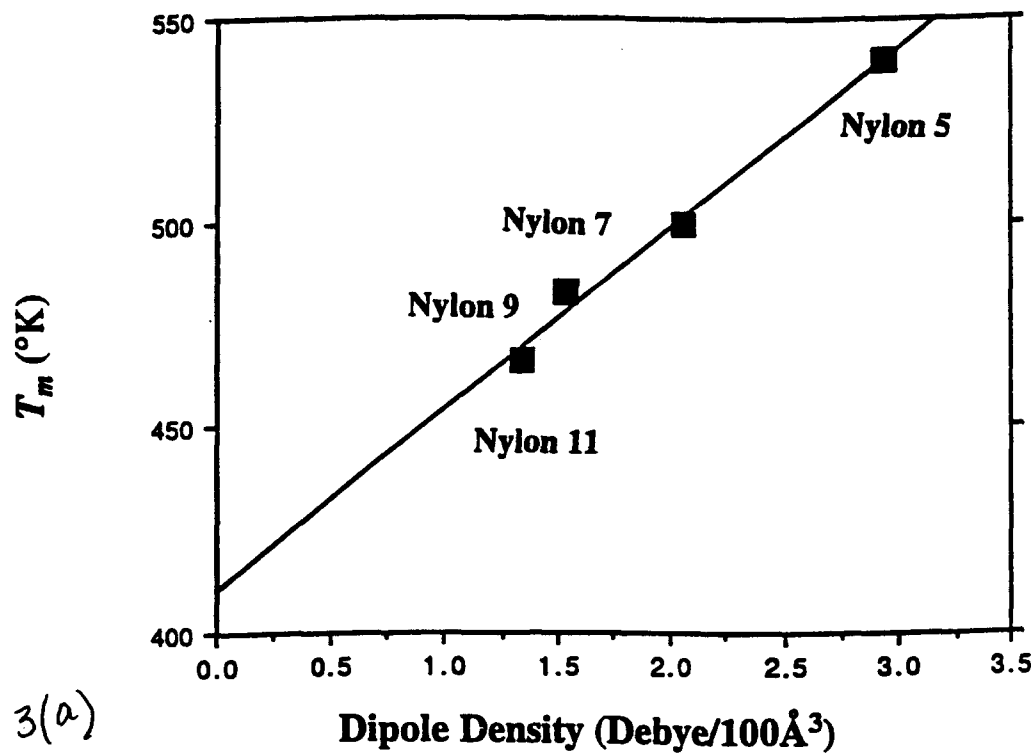


Fig 3(a)

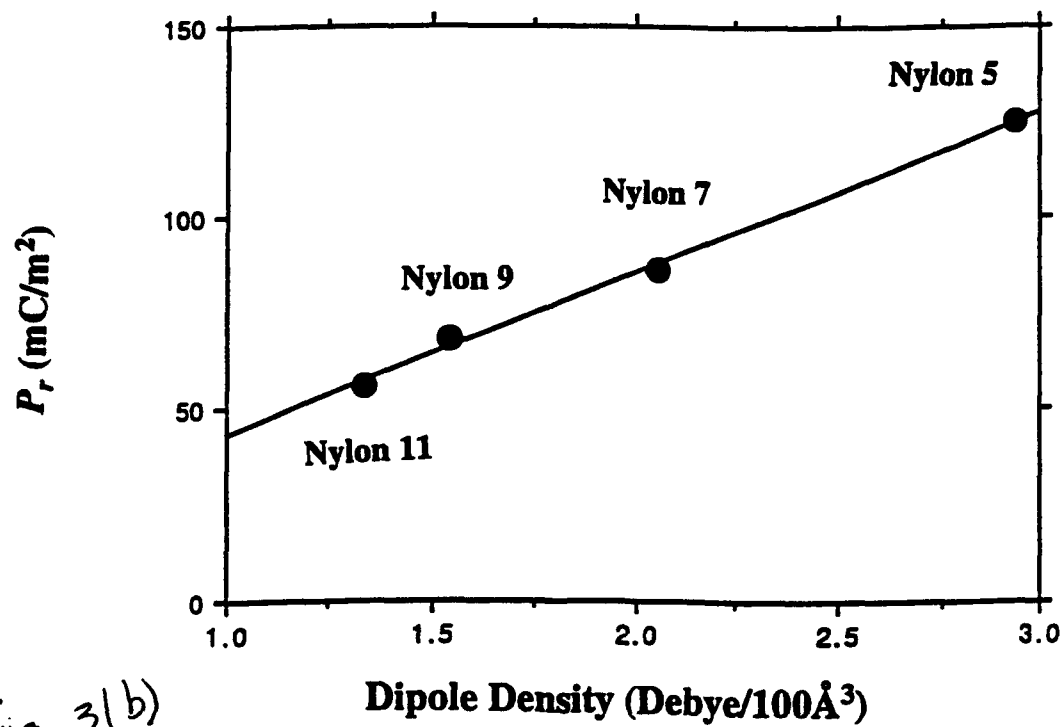


Fig. 3(b)

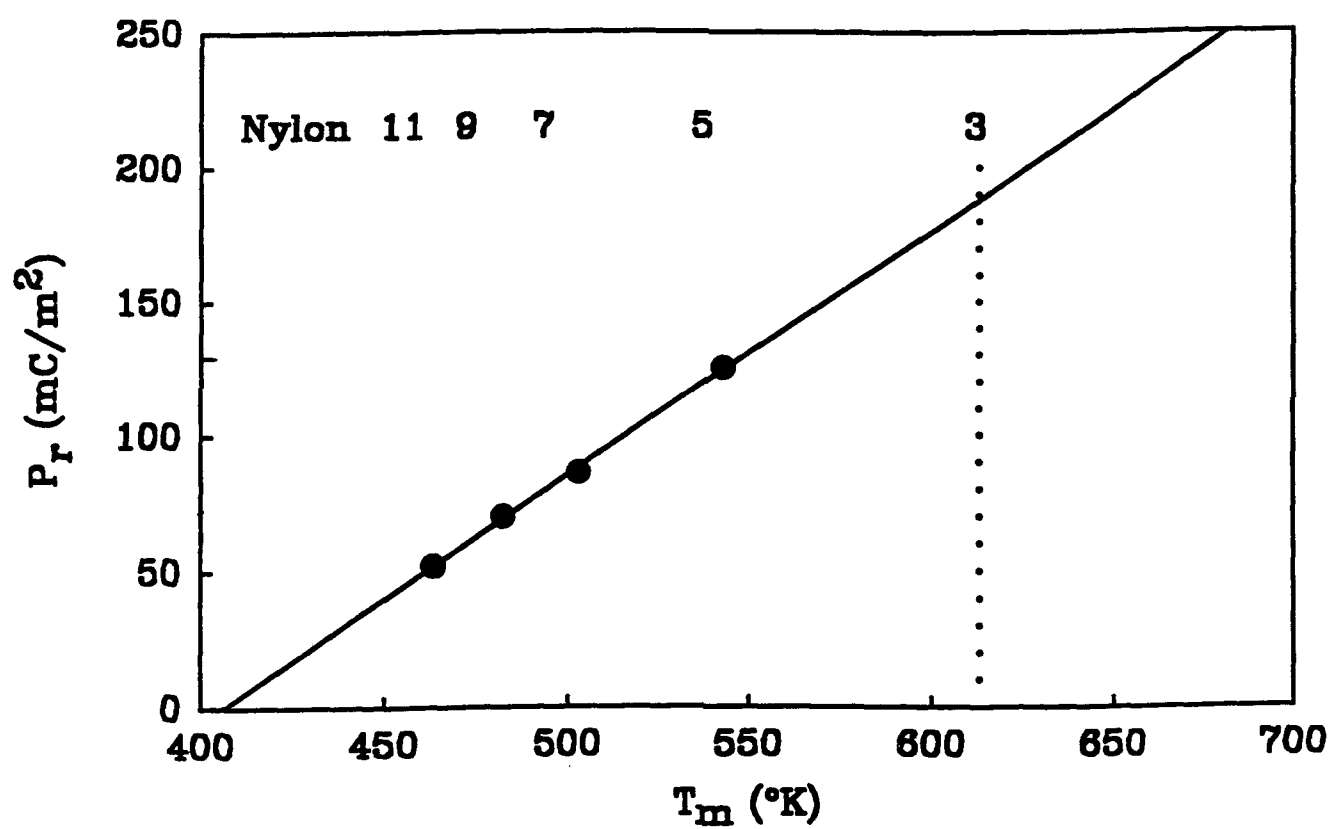


Fig. 4

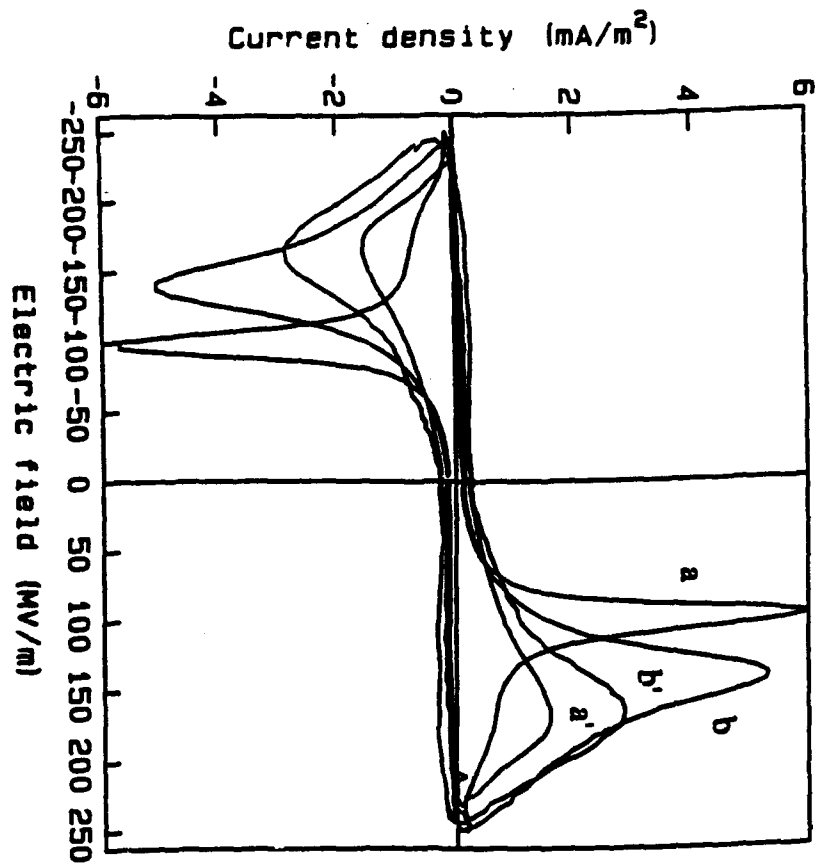


Fig 5(a)

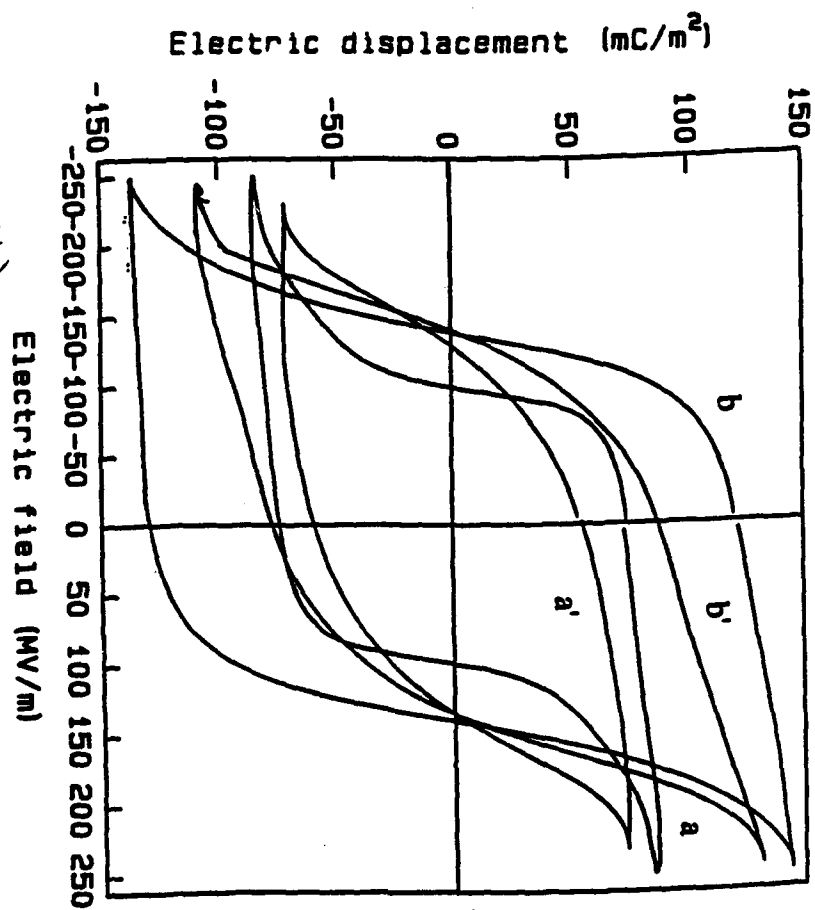


Fig 5(b)

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